TMR 190

DESIGN STUDY ON AN ALTERNATE METHOD FOR PRODUCTION OF AMMONIUM PERCHLORATE (U)

17 July 1961

U. S. NAVAL PROPELLANT PLANT, INDIAN HEAD, MARYLAND

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by

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FOREWORD

The study presented here was completed in mid-1958. Therefore, the price information is somewhate out of date, but a quick review of current information indicates that changes in cost of ingredients and byproducts are favorable to the proposed process.

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Technical Director

Released by:

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ABSTRACT

Preparation of ammonium perchlorate by double decomposition of ammonium sulfate and sodium perchlorate was used to determine a target price for economic production.

DESIGN STUDY ON AN ALTERNATE METHOD FOR PRODUCTION OF AMMONIUM PERCHLORATE

The increased importance of ammonium perchlorate as a component of missile propellants makes it advisable to review the techniques of manufacture not only to establish alternate sources but also to establish a reasonable economic target price. This report includes a design study and cost estimates for preparation of ammonium perchlorate by a double decomposition reaction between sodium perchlorate and ammonium sulfate. The electrolytic process for production of sodium perchlorate from sodium chlorate offers little opportunity for improvement.

In the most widely used commercial process, sodium perchlorate is reacted with ammonia and hydrochloric acid and the ammonium perchlorate and sodium chloride are then removed by fractional crystallization. This process has several disadvantages:

(1) The hot chloride solution in the presence of oxidizers causes excessive stress corrosion cracking and general corrosion of the process vessels;

(2) Problems in metering the hydrochloric acid and ammonia make it difficult to maintain optimum pH;

(3) Variation in pH makes it necessary to recycle as much as 20% to 40% of the product before it will meet specifications.

The ammonium perchlorate produced in this type of operation costs from $0.34 to $0.40 per pound.

PROPOSED ALTERNATE PROCESS

The double decomposition reaction between ammonium sulfate and sodium perchlorate was selected for study because of the widespread availability of ammonium sulfate. Furthermore, information is available on the equilibria of solutions resulting from this reaction. Freeth[(1)] showed ammonium perchlorate and sodium sulfate can be prepared by reacting equivalent weights of sodium perchlorate and ammonium sulfate in a mother liquor consisting of either.

Sodium sulfate can then be recrystallized from the hot and ammonium perchlorate from the cold remainder.

Equipment required includes a sodium perchlorate-ammonium sulfate dissolver, sodium sulfate vacuum crystallizer, and ammonium perchlorate cooling crystallizer. Solids feeders for the dissolver and a centrifuge and dryer for each crystallizer are also required. Figure 1 is a flow chart for a 10-ton-per-day ammonium perchlorate plant showing equipment, flow rates, and compositions. This design can be scaled directly.

Ammonium sulfate and sodium perchlorate are added to the dissolver which is maintained at 65° C to ensure solution of the reactants in the recycled mother liquor. The effluent passes to the sodium sulfate crystallizer. By application of heat and constant vacuum, process water is removed and the sodium sulfate is crystallized. Sodium sulfate is then removed in a slurry from the suspension container of the crystallizer, dewatered, washed, and dried. The effluent at 60° C passes to the ammonium perchlorate crystallizer. After the solution has been cooled to 25° C, ammonium perchlorate is selectively crystallized. The product is then dewatered, washed, and dried. Mother liquor from the ammonium perchlorate crystallizer is returned to the dissolver for reuse.

Material Balance

Based on a closed-cycle continuous process, the following liquor concentrations were selected:

	Dissolver effluent (%)	Sodium sulfate crystallizer effluent (%)	Ammonium perchlorate crystallizer effluent (%)
Na_2SO_4	23.4	18.4	20.6
NH_4ClO_4	15.1	16.1	6.0
$(NH_4)_2SO_4$	10.0	10.7	12.0
H_2O	51.5	54.8	61.4

The material balance is based on the following equation:

$$\underset{132.14}{(NH_4)_2SO_4} + \underset{2(122.45)}{2NaClO_4} \rightarrow \underset{2(117.50)}{2NH_4ClO_4} + \underset{142.05}{Na_2SO_4}$$

NH_4ClO_4 – 10T/day
– 834 lb/hr
– 13.9 lb/min

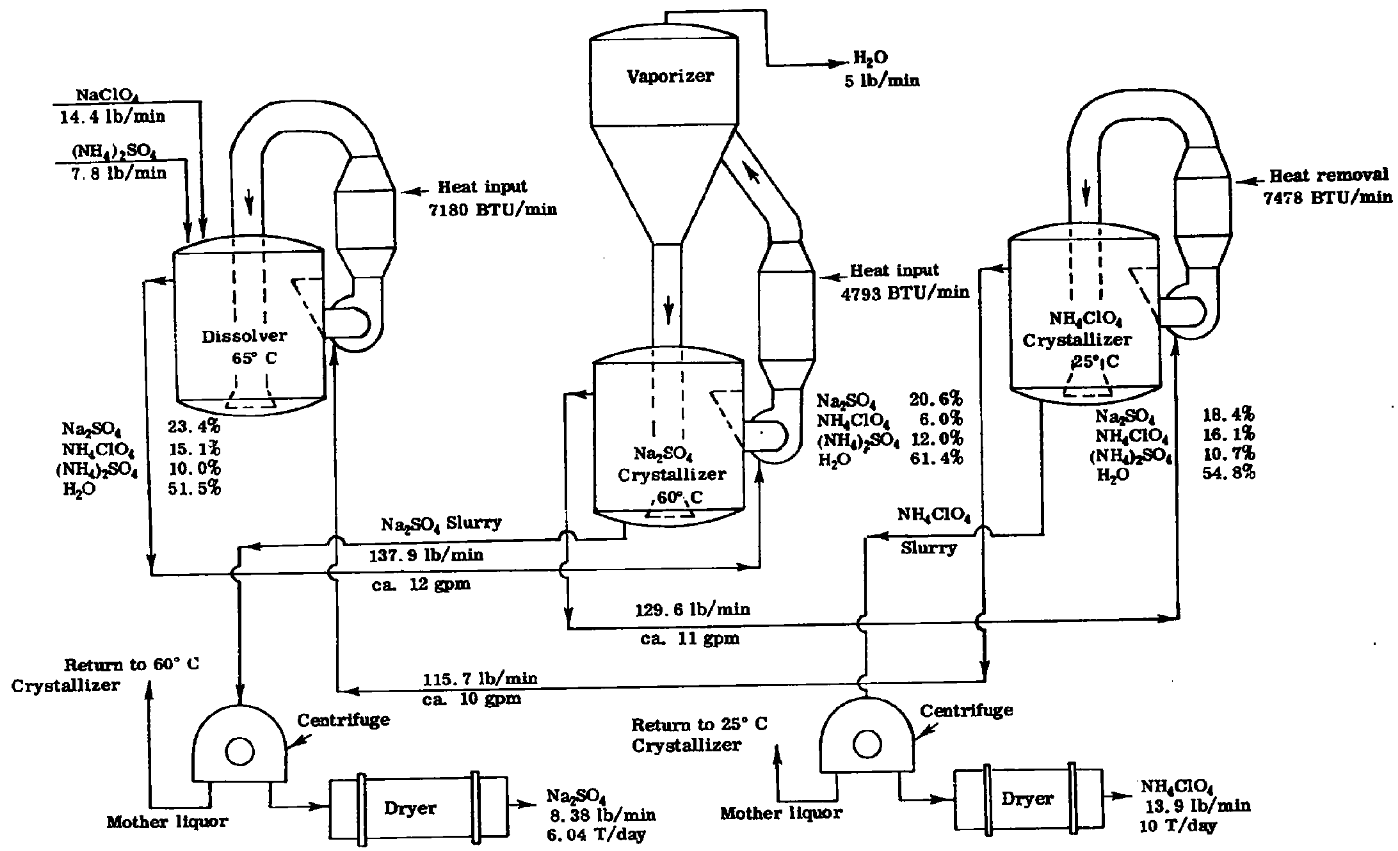

FIGURE 1. FLOW CHART FOR THE PRODUCTION OF AMMONIUM PERCHLORATE (10 T/DAY)

Na_2SO_4	→ 6.04 T/day
	→ 503 lb/hr
	→ 8.38 lb/min
$(NH_4)_2SO_4$	→ 5.62 T/day
	→ 468 lb/hr
	→ 7.8 lb/min
$NaClO_4$	→ 10.42 T/day
	→ 868 lb/hr
	→ 14.4 lb/min

The flow rates were determined from the solution compositions to provide a production rate of 10 T/day ammonium perchlorate.

$(NH_4)_2SO_4$	to dissolver	7.8 lb/min at STP
$NaClO_4$	to dissolver	14.4 lb/min at STP
Mother liquor	to dissolver	115.7 lb/min at 25° C
Solution	to Na_2SO_4 crystallizer	137.9 lb/min at 65° C
Solution	to NH_4ClO_4 crystallizer	129.6 lb/min at 60° C
Na_2SO_4	produced	8.38 lb/min
NH_4ClO_4	produced	13.9 lb/min

Heat Balance

The heat balance for this process was accomplished by taking each unit in this closed system and analyzing the heat involved. Assumptions were made as to the specific heat of the solutions, radiation and convection losses, and heat required to evaporate water introduced as centrifuge wash.

Dissolver:

The heat balance on the dissolver is based on taking 115.7 lb/min of mother liquor at 25° C and heating to 65° C and concurrently dissolving 7.8 lb/min of ammonium sulfate and 14.4 lb/min of sodium perchlorate. The resulting solution of 137.9 lb/min at 65° C is sent to the sodium sulfate crystallizer.

Heat of Reaction: The standard heat of reaction at 25° C was calculated for the following reaction:

$$(NH_4)_2SO_{4(c)} + 2NaClO_{4(c)} \rightarrow 2NH_4ClO_{4(solu)} + Na_2SO_{4(solu)}$$

$$(NH_4)_2SO_{4(c)}\Delta H = -Q_f = -13{,}500 \text{ CHU } (7.8 \text{ lb})$$

$$2NaClO_{4(c)}\Delta H = -Q_f = -24{,}600 \text{ CHU } (14.4 \text{ lb})$$

$$2NH_4ClO_{4(solu)}\Delta H = -Q_f = -18{,}500 \text{ CHU } (13.9 \text{ lb})$$

$$Na_2SO_{4(solu)}\Delta H = -Q_f = -19{,}800 \text{ CHU } (8.4 \text{ lb})$$

$$-13{,}500 - 24{,}600 = -18{,}500 - 19{,}800 + Q_R$$

$$Q_R = 200 \text{ CHU}$$

$$\text{Heat of reaction} = 360 \text{ BTU/min}$$

Sensible Heat: Heat required to raise temperature of solution, including reactant, from 77° F to 149° F (Δt = 72° F).

$$Q_S = WC_p\Delta t$$

$$Q_S = 137.9 \text{ lb/min} \times 0.75 \times 72° \text{ F}$$

$$Q_S = 7447 \text{ BTU/min}$$

Heat Loss: In order to estimate the heat loss it is necessary to establish the approximate size of the dissolver. Sodium sulfate has an inverted solubility. In order to prevent salting out on the heater surface, the maximum temperature rise should not be more than 1°-3° F. This would require a circulating system of about 600-700 gpm with the dissolver holding about 2000 gal. With about 250 sq ft of surface and 2 in. of insulation, the heat loss is estimated.

$$R = \frac{X}{KA} = \frac{2/12}{0.0418 \times 250} = 0.016$$

$$Q_1 = \frac{\Delta t}{R} = \frac{90}{0.016} = 5625 \text{ BTU/hr} = 93 \text{ BTU/min}$$

Heat Requirements (dissolver):

$$Q = Q_R + Q_S + Q_1$$
$$Q = 360 - 7447 - 93$$
$$Q = -7180 \text{ BTU/min}$$
$$= -430{,}800 \text{ BTU/hr}$$

Sodium Sulfate Crystallizer:

The heat balance on this crystallizer is based on introducing 137.9 lb/min of solution at 65° C, supersaturating the solution by

vacuum evaporation and cooling the solution to 60° C. Sodium sulfate will be crystallized at the rate of 8.38 lb/min. The overflow of the crystallizer, 129.6 lb/ min at 60° C, passes to the ammonium perchlorate crystallizer. In addition to the heat of crystallization and sensible heat in the feed solution, the heat losses of radiation and convection and heat required to evaporate excess process water are considered.

Heat of Crystallization:

$$Q_c = H_c \frac{W}{M} = -358 \frac{8.38}{142.05}$$

$$Q_c = -21 \text{ CHU}$$

$$Q_c = -38 \text{ BTU/min}$$

Sensible Heat:

$$Q_S = C_p W \Delta t$$

$$= 0.75 \times 137.9 \text{ lb/min} \times 5 \times 1.8$$

$$= 931 \text{ BTU/min}$$

Heat Loss: is assumed to be twice as large as it is in the dissolver since vapor body and suspension container are involved.

$$Q_1 = 186 \text{ BTU/min}$$

Process Water Evaporation: It is assumed that about 5 lb/min of water will be required to be evaporated. This amount will be introduced through centrifuge washer and pump seals.

$$Q_e = H_w = 1100 \times 5$$

$$Q_e = 5,500 \text{ BTU/min}$$

Heat Requirements: (Na_2SO_4 Crystallizer)

$$Q = Q_c + Q_S + Q_1 + Q_e$$

$$Q = -38 + 931 - 186 - 5500$$

$$Q = -4793 \text{ BTU/min}$$

Ammonium Perchlorate Crystallizer:

The heat requirements for this crystallizer are based on taking 129.6 lb/min of liquor at 60° C, cooling this liquor in the crystallizer

to 25° C, and crystallizing ammonium perchlorate. The balance of 115.7 lb/min of mother liquor at 25° C will be returned to the dissolver.

Heat of Crystallization:

$$Q_c = H_c \frac{W}{M} = 6360 \times \frac{13.9}{117.5}$$
$$Q_c = 752 \text{ CHU}$$
$$Q_c = 1354 \text{ BTU/min}$$

Sensible Heat:

$$Q_S = C_p W \Delta T$$
$$= 0.75 \times 129.6 \times 35 \times 1.8$$
$$= 6124 \text{ BTU/min}$$

Heat Loss: Heat loss at 25° C is negligible.

Total Heat:

$$Q = Q_c + Q_S$$
$$Q = 1354 + 6124$$
$$Q = 7478 \text{ BTU/min}$$

ADVANTAGES

The process proposed here will simplify the selection of materials of construction and the maintenance of equipment. Rubber-lined tanks for hydrochloric acid, pressure storage tanks for ammonia, and high-vacuum equipment for ammonium perchlorate crystallization will not be necessary. The glass-lined reactor required for addition of ammonia and hydrochloric acid will also not be necessary.

With the proposed process, a standard 304 or 316 stainless steel should be adequate. From the standpoint of economic considerations and plant layout, further refinements in the process might be made. The water used to dissolve the sodium perchlorate for transport to the processing area might be eliminated; the mother liquor might be substituted for the water but this has some disadvantages.

MANUFACTURING COSTS

The purpose of this section is to discuss the cost of ammonium perchlorate. By analysis of the market costs of various chemicals used in the existing, proposed, and related processes, the expected manufacturing costs have been determined.

Basic Cost:

These data are based on prices taken from Hi-Lo Chemical Price Issue of the Oil, Paint, and Drug Reporter.

Chemical	Price/lb	Price/ton
Sodium chlorate	$0.09	
Potassium chlorate	0.1125	
Potassium chloride	0.014	$28
Sodium chloride	0.005	10
Potassium perchlorate	0.1850	..
Ammonia	0.036	72
Muriatic acid (20° Be)	0.015	30
Sodium sulfate	0.026	52
Ammonium sulfate	0.021	42

Processing Cost:

The object of this part is to determine the processing cost for converting a salt by double decomposition reaction accompanied by crystallization. The following reaction was selected as typical of this type of process:

$$\underset{0.87\ \text{lb}}{NaClO_3} + \underset{0.61\ \text{lb}}{KCl} \rightarrow \underset{0.48\ \text{lb}}{NaCl} + \underset{1\ \text{lb}}{KClO_3}$$

Processing cost is determined by subtracting the credit for sodium chloride produced from the ingredient cost and, in turn, subtracting this ingredient cost figure from the cost of the product.

$$\begin{array}{llr}
NaClO_3 & = 0.87 \times .09 & = 0.0783 \\
KCl & = 0.61 \times .014 & = \underline{0.0085} \\
 & & 0.0868 \\
NaCl & = 0.48 \times .005 & = \underline{0.0024} \\
 & & 0.0844 \\
KClO_3 & = 1 \quad \times .1125 & = 0.1125 \\
 & & -\ \underline{0.0844} \\
\text{Processing Cost} & & 0.0281
\end{array}$$

Therefore, the processing cost in this type of process is estimated to be about $0.03 per pound of product.

Sodium Perchlorate:

This chemical is not an article of trade. No published price exists. It is necessary to estimate the cost for sodium perchlorate by using the following process:

$$\underset{0.89\ \text{lb}}{NaClO_4} + \underset{0.54\ \text{lb}}{KCl} \longrightarrow \underset{0.42\ \text{lb}}{NaCl} + \underset{1\ \text{lb}}{KClO_4}$$

By using the processing cost previously determined, the cost of sodium perchlorate is determined.

Processing cost	= 0.0300
KCl = 0.54 × .014	= 0.0076
	0.0376
Less NaCl credit (0.42 × .005)	= 0.0021
Ingredient and process cost	0.0355
$KClO_4$ = 1 × .1850	= 0.1850
Less ingredients and processing	= 0.0355
$NaClO_4$ (0.89 lb)	0.1495
	= 0.1680/lb

Therefore, sodium perchlorate is estimated at a unit cost of $0.1680 per pound. Considering that sodium chlorate costs $0.09 per pound, the spread of about $0.08 per pound of sodium perchlorate is adequate to allow converting sodium chlorate to sodium perchlorate.

Proposed Process:

The cost of ammonium perchlorate produced by the proposed process would be as follows:

$$\underset{0.56\ \text{lb}}{(NH_4)_2SO_4} + \underset{1.04\ \text{lb}}{2NaClO_4} \longrightarrow \underset{0.60\ \text{lb}}{Na_2SO_4} + \underset{1\ \text{lb}}{2NH_4ClO_4}$$

$(NH_4)_2SO_4$ = 0.56 × 0.021	= 0.0118
$NaClO_4$ = 1.04 × 0.1680	= 0.1747
Processing	= 0.0300
	0.2165
Less Na_2SO_4 credit	0.0159
NH_4ClO_4 cost	0.2006

<u>Existing Process:</u>

A comparable cost for manufacture of ammonium perchlorate by an existing process is shown as follows:

$$\begin{array}{ccccc} 0.31\text{ lb} & 0.14\text{ lb} & 1.041\text{ lb} & 0.5\text{ lb} & 1\text{ lb} \\ HCl & +\ NH_3 & +\ NaClO_4 & \rightarrow\ NaCl & +\ NH_4ClO_4 \end{array}$$

HCl	$= 0.31 \times \frac{1}{.3145} \times 0.015$	$= .0148$
NH_3	$= 0.14 \times .036$	$= .0054$
$NaClO_4$	$= 1.04 \times .1680$	$= .1747$
Processing		$= .0300$
		$.2249$
Less NaCl credit		$= .0025$
NH_4ClO_4 cost		$.2224$

TARGET PRICE

The proposed process is about \$0.022 per pound less expensive than the existing process. Therefore, the real target price for ammonium perchlorate is about \$0.20 per pound. This represents a potential saving to the government of \$0.14 to \$0.20 per pound based on the current price of \$0.34 to \$0.40 per pound. On the basis of 1,000,000 pounds per month, this amounts to an annual saving of from \$1,680,000 to \$2,400,000 per year. The cost involved in obtaining a specific particle size distribution has not been considered.

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